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SOLVENT EXTRACTION OF URANIUM FROM A CHLORIDE LEACH LIQUOR

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by

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ABSTRACT

Conventional processing of uranium ores involves leaching with either sulphuric acid or alkaline carbonate solutions. Both processes, although effective for uranium, result in tailings containing essentially all the radionuclides such as Ra^{226} and Th^{230}. The paper describes an alternative process flowsheet whereby the ore or concentrate is leached in hydrochloric acid, solubilizing all the radionuclides. The uranium is recovered by solvent extraction with a 2.5% solution of TOPO in Shell 140 diluent and the solvent is scrubbed to remove impurities using a uranium chloride solution. Because TOPO is difficult to strip for uranium recovery, a new stripping technique was developed using a solution mixture consisting of 5% H_2SO_4 + 5% H_2O_2. Finally the stripped solvent is scrubbed with 2 M NaCl to remove sulphate prior to recycling the solvent to the extraction circuit.

PROCÉDÉ D'EXTRACTION PAR SOLVANT POUR RÉCUPÉRER L'URANIUM CONTENU DANS UNE SOLUTION DE CHLORURE

par

G.M. Ritcey et E.W. Wong

RÉSUMÉ

Les méthodes de traitement classiques des minerais d'uranium comprennent les procédés de lixiviation avec soit des solutions d'acide sulfurique ou de carbonate alcalin. Les deux procédés, bien qu'efficaces pour l'uranium, produisent des résidus contenant essentiellement tous les radionucléides tels que Ra^{226} et Th^{230}. Le présent rapport décrit un schéma de procédé de rechange dans lequel le minerai ou le concentré est lixivié dans de l'acide hydrochlorique, solubilisant tous les radionucléides. L'uranium est récupéré à l'aide d'un procédé d'extraction par solvant avec une solution à 2,5 % de TOPO dans le diluant Shell 140, et le solvant est épuré afin d'enlever les impuretés au moyen d'une solution de chlorure d'uranium. En raison de la difficulté d'effectuer l'épuration du TOPO pour récupérer l'uranium, une nouvelle technique d'épuration a été mise au point à l'aide d'un mélange d'une solution composée de 5 % H_2SO_4 + 5 % H_2O_2. Enfin, le solvant épuré est lavé de nouveau avec 2 M NaCl afin d'enlever le sulfate avant de recycler le solvant dans le circuit d'extraction.

Introduction

The processing of uranium ores throughout the world has relied for the most part on leaching of the crushed and ground ore in sulphuric acid. Only a few plants have used alkaline leaching. Purification of the sulphuric acid leach liquor is by solvent extraction or ion exchange or a combination of both processes, followed by precipitation of the uranium from the purified solution to produce yellowcake. Though commercially successful, the process leaves nearly all the radionuclides (Ra^{226}, Pb^{210}, Po^{210}, Th^{230}, etc.) in the tailings.

During the period 1977-1984 new alternative technology has been developed at CANMET, with the major objectives being to reduce the environmental impact, improve uranium recovery, improve by-product recovery, and to maintain an economically viable process.

Because H_2SO_4 leaching does not solubilize many of the radionuclides[1], other possible process circuits were investigated. An examination of the HNO_3 route showed that most of the radionuclides were solubilized from conventional low grade ores, but the solubility of Ra^{226} was poor when treating complex uranium-nickel-arsenide ores and the tailings still contained greater than 100 pCi/g Ra^{226}[2]. Subsequent studies included the possibility of using the chloride ion to provide radionuclide solubility. Two routes were developed concurrently; one a chlorination roast followed by leaching[3], and the other a leach in HCl[4].

In the treatment of the conventional low grade ores, of the Elliot Lake type in Canada, both chloride processes were technically feasible, in that a tailings resulted with 25 pCi/g Ra^{226}. Also, increased solubility of thorium and rare earth occurred as well as an increased dissolution of uranium.

Concurrent with the development of leaching technology, investigations were in progress on the possible application of solvent extraction for the separation of uranium, thorium and rare earths from the leach liquor. This paper describes a process that was developed for the recovery of uranium from the chloride solution by solvent extraction.

Procedure

As was stated in the introduction, the chloride solutions containing uranium were derived from the leaching of uranium ores by either HCl leaching or by a chlorination roast followed by dilute HCl leaching. The respective flowsheets are shown in Fig. 1 and Fig. 2 (details of the circuits are given in the referenced papers). The typical leach liquor composition resulting from the chloride leaching of Elliot Lake ores (Table 1) is given in Table 2.

Table 1

Typical Elliot Lake Ore Analysis

	%
U	0.14
Th	0.036
Fe	2.77
S	2.87
Si	38.4
Al	3.6
Total rare earths	0.15
Ra	400 pCi/g

Table 2

Leach Solution Analysis
g/L

U	0.65
Th	0.20
Fe^{T}	3.09
Fe^{2+}	2.33
Si	0.075
Free HCl	14.4
Ra^{226}	3720 pCi/L

Following preparation of the chloride leach feed solution to solvent extraction, various selected extractants were screened for possible application to the extraction of uranium. The extractants were usually 5% by volume initially in Solvesso 150 aromatic diluent which was subsequently

changed to Shell 140 aliphatic diluent in order to improve the phase disengagement rates. The initial screening tests were conducted on synthetic feed solutions shown in Table 3.

Table 3

Synthetic Feed Solution
g/L

	U	Fe^{3+}	Mg	Al	Th	Y	Na	Ca
No. 1	2.0	2.0	2.0	2.0	-	-	-	-
No. 2	0.1	1.0	1.0	0.5	1.0	1.0	1.0	1.0

Following the screening tests, the flowsheet was subsequently developed using leach feed as described in Table 2, in contact with the extractant. Conditions were optimized and a continuous flow circuit then run to verify the parameters.

Results

Screening Tests on Selected Extractants

Screening tests were performed on selected extractants as shown in Table 4[5]. The conditions of these tests, using 5 vol % of the extractant in kerosene was to contact the solvent mixture with an equal volume of synthetic feed solution for 5 minutes at room temperature.

The data in Table 4 indicated the following:

1) TOPO was the best for extraction of uranium for the range of low acidity (pH 2) to high acidity (6M HCl). Increase in chloride concentration from 0 to 3M had little or no effect on the uranium extraction, compared to greater effects with the other extractants.

2) At low acidities (pH 1-2) and no NaCl addition DEHPA extracted uranium completely but extraction decreased with increasing NaCl added.

3) An acidity of 2-6M HCl was required for Aliquat 336 and NaCl concentration seemingly had no effect.

4) An acidity of 6M was required for fair extraction with either Alamine 336 or TBP. The extraction increased with increase in NaCl concentration for each extractant.

5) Primene JMT and Adogen 283 were poor and inferior to all the above extractants.

6) The order of extractability of uranium noted for these extractants was as follows:

TOPO > DEHPA > quaternary amine > tertiary amine > TBP secondary amine > primary amine

Table 4

Screening for Uranium Extractants

Reagent	Molarity NaCl (M)	Extraction (%) Acidity (M) HCL				
		pH 2	pH 1	2M	4M	6M
TOPO	0	25.9	95.0	96.2	100	99.9
DEHPA		99.3	98.3	76.5		7.0
Alamine 336		7.5	4.4	20.2	66.5	85.9
Aliquat 336		50.0	57.2	99.3	99.9	
Primene JMT		0.55	0.2	2.2	0.89	7.0
Adogen 283		2.0	1.7	9.8	32.4	43.8
TBP		0.58	0.54	14.2	72.1	96.8
TOPO	1.0		99.8	92.6	85.3	
DEHPA			98.1	65.3	19.1	
Alamine 336				31.6	57.8	
Aliquat 336			94.6	95.3	85.8	
Primene JMT				3.7	9.8	
Adogen 283				15.8	29.4	
TBP			2.34	32.6	76.5	
TOPO	3.0			93.0		
DEHPA				37.6		
Alamine 336				64.5		
Aliquat 336				98.4		
Primene JMT				1.6		
Adogen 283				26.9		
TBP				72.0		

Feed = Synthetic Solution No. 1

The second series of screening tests, using the same reagents, involved contact with a weak solution of uranium, but one which contained relatively high concentrations of impurities (the analysis of No. 2 synthetic feed is given in Table 3). The results of the tests, in summary are:

1) TOPO extracted U over the range pH 2 - 6M HCl and also co-extracted thorium. Very little Y was extracted.

2) DEHPA extracted U at pH 1-2, Th over the range pH 2 - 6M HCl. Very little Y extracted.

3) Aliquat 336 extracted U at 2-6M HCl with no NaCl addition, and over the range pH 2 - 4M by the addition of 1-3M NaCl. Very little Th or Y extracted.

4) Alamine 336 was similar to Aliquat 336 but slightly less effective for the extraction of U.

5) TBP was effective for U extraction only at 6M HCl or 1M HCl + 3M NaCl. Very little Th was extracted, and about 1% Y extracted.

A summary of the TOPO data is shown in Table 5. Because of the overall performance of TOPO, it was selected as the extractant for uranium from chloride solutions.

The initial tests used the aromatic diluent Solvesso 150 for the TOPO. However the subsequent preliminary work with actual feed solution resulted in long phase separation time and very thick emulsion layers at the interface. By changing to the aliphatic diluent Shell 140 there was a significant improvement. To further improve the separation, 30 ppm Polyox flocculant was added to the feed solution. This resulted in the precipitation of a large amount of white gel, composed mainly of silica.

Table 5

Extraction with TOPO for U, Th, Y

Element	Molarity NaCl	Extraction (%) Acidity (M) HCl				
		pH 2.0	pH 1.0	2M	4M	6M
U	0	90.9	99.9	94.9	99.0	99.9
	1.0	97.7	90.9	87.6	78.6	
	3.0			99.9		
Th	0	95.3	89.7	93.7	91.3	98.8
	1.0	93.4	88.5	91.5	96.3	
	3.0			97.5		
Y	0	0.73	0.73	0.82	0.60	0.58
	1.0	0.062	0.4	0.45	0.58	
	3.0			0.60		

Feed = Synthetic Solution No. 2

Selection of TOPO Concentration for Extraction

Two concentrations of TOPO were compared for uranium loading, discrimination over Th and Fe, and equilibrium curves. The tests were performed on 2.5 wt. % and 5 wt. % TOPO in Shell 140, at varying phase ratios (A/O), in contact for 5 minutes with the leach feed solution at room temperature.

In Tables 6 and 7 are given the extraction data for the two solvents. In Table 8 is shown the comparative extractions and separation factors for U/Th, U/Fe for the two solvent mixtures. As would be expected the stronger solvent loaded more uranium than the weaker TOPO. However the 2.5% TOPO was considerably more selective over Th and Fe during the extraction stage. The equilibrium isotherms are given in Fig. 3.

From the above data it was concluded that 2.5% TOPO was a sufficient concentration for such a system where the feed solution was about 0.65 g U/L and 2.5% TOPO had a loading capacity of about 3 g U/L. Perhaps even more important, 2.5% TOPO, because it was more selective over Th and Fe co-extraction would therefore be possibly more amenable to scrubbing. Therefore 2.5% TOPO was selected for all subsequent tests.

The McCabe-Thiele construction is shown in Fig. 4, indicating 4-5 stages would be required for extraction from a feed solution containing 0.65 g U/L to obtain a raffinate of 0.003 g U/L.

Table 6

Extraction by 5% TOPO in Shell 140

A/O	Raffinate (g/L)			Solvent (g/L)			Extraction (%)		
	U	Th	Fe	U	Th	Fe	U	Th	Fe
20/1	0.96	0.20	3.08	7.29	0.009	0.27	55.2	0.225	0.44
15/1	0.27	0.20	3.07	5.83	0.015	0.34	58.9	0.50	0.73
10/1	0.15	0.20	3.05	5.05	0.024	0.39	76.5	1.20	1.2
5/1	0.12	0.19	3.00	2.70	0.041	0.45	81.8	4.1	2.9
3/1	0.09	0.18	2.95	1.70	0.050	0.43	85.9	8.3	4.6
1/3	0.0017	0.068	2.52	0.22	0.044	0.19	99.7	66.0	18.4
1/5	0.0018	0.005	2.44	0.11	0.030	0.13	99.7	75.0	21.0

Table 7

Extraction by 2.5% TOPO in Shell 140

A/O	Raffinate (g/L)			Solvent (g/L)			Extraction (%)		
	U	Th	Fe	U	Th	Fe	U	Th	Fe
20/1	0.50	0.20	3.09	3.27	0.004	0.06	24.8	0.10	0.097
10/1	0.36	0.20	3.08	3.02	0.007	0.07	45.8	0.35	0.24
5/1	0.21	0.20	3.07	2.24	0.011	0.09	67.8	1.1	0.60
3/1	0.06	0.20	3.05	1.79	0.017	0.12	90.4	2.8	1.29
1/3	0.005	0.12	2.73	0.22	0.026	0.12	100	39.0	11.7
1/5	0.003	0.057	2.63	0.10	0.021	0.09	75.8	52.5	15.0

Table 8

Extractant Concentration and Discrimination
(Feed 0.66 g U/L)

TOPO %	A/O Ratio	Raff. g U/L	Loaded Solvent (g/L) U	Fe	Th	Discrimination U/Th	U/Fe
2.5	20/1	0.49	3.27	0.06	0.004	817	54
	10/1	0.35	3.02	0.07	0.007	431	42
	5/1	0.20	2.24	0.09	0.011	203	24
	3/1	0.063	1.79	0.12	0.017	105	15
	1/3	0.005	0.22	0.012	0.026	8.5	1.8
	1/5	0.003	0.10	0.09	0.021	4.7	1.1
5.0	20/1	0.29	7.29	0.27	0.009	810	27
	15/1	0.27	5.83	0.34	0.015	389	17
	10/1	0.15	5.05	0.39	0.024	210	13
	5/1	0.12	2.70	0.43	0.041	17	6
	3/1	0.017	1.70	0.19	0.05	14	3.9
	1/5	0.0018	0.11	0.13	0.03	3.0	0.8

Scrubbing of the Solvent to Remove Impurities

The extraction data on TOPO concentration indicated that although the 2.5% TOPO was reasonable in its discrimination over Th and Fe and better than the 5.0% TOPO, there was nevertheless some co-extraction of these impurities. Various scrub solutions were considered, and the solution selected was 15 g U/L in chloride medium at pH 0.5 HCl. This uranium chloride scrub solution was chosen based on previous work in other solvent extraction systems by the authors, where the scrub solution consisted of the metal being extracted (however a uranyl sulphate scrub could also be used). The data for such a scrub solution are shown in Table 9, where the loaded solvent was contacted with the scrub solution for 5 minutes at an O/A ratio of 3.

Table 9

Scrubbing for Removal of Thorium and Iron

	Org Scrub Feed	Scrubbed Org.	Scrub Feed	Scrub Liquor	SF++ U/Metal
U (g/L)	2.61	5.47	14.45	5.05	-
Fe (g/L)	0.071	< 0.003	0	0.28	> 1800
Th (g/L)	0.011	< 0.003	0	0.080	> 1800
Free HCL (g/L)	-	-	33.9	29.5	-
Y (g/L)	-	-	ND*	ND	-
Ra^{226} pCi/L	-	-	-	2960	-

*Not detected
++Separation Factor, U/Fe, U/Th

Stripping of Uranium from TOPO

Although the TOPO was a good extractant for uranium from chloride solutions, it proved difficult to strip. A considerable number of stripping tests were performed to qualitatively determine whether stripping occurred and to examine the physical characteristics of phase separation. The data from the initial screening of reagents are shown in Table 10. Of the possible stripping agents examined, only H_2SO_4+H_2O_2 gave any visual indication of uranium being stripped. No emulsions were formed as had occurred in most of the other tests, and the phase disengagement time was very fast at 11 seconds. The concentration of H_2SO_4 was varied at 5% and 10% while maintaining 5% H_2O_2 to compare the relative stripping effectiveness, but there was no significant difference. All subsequent work was with the 5% H_2SO_4 + 5% H_2O_2 mixture for stripping. In Fig. 5 is shown a stripping isotherm, at 5 minutes contact time using that strip mixture. About seven theoretical stages at about O/A 2/1 are indicated to achieve about 0.5 g/L in the stripped solvent. In Table 11 are given the strip data indicating the relative effectiveness of the stripping agent with respect to discrimination over Th and Fe. Although the Fe stripping percentage was high, it should be noted that the actual Fe concentration in the solvent was low to begin with (0.007 g Fe/L) due to effective scrubbing.

Scrubbing for Sulphate Removal

The problem with stripping in a sulphate medium was that sulphate loaded onto the organic and this was undesirable for two reasons. Firstly, the sulphate occupied sites on the organic, thus decreasing the extraction efficiency of the TOPO as it was recycled. Secondly, the sulphate would also allow Ra^{2+} to precipitate as $RaSO_4$ when recycling the chloride raffinate. Screening of scrubbing reagents indicated that several contacts with 2M NaCl would effectively scrub the majority of the sulphate off the TOPO. The data in Table 12 show the results of scrubbing a 2.5% TOPO solvent that had been partially stripped with 5% H_2SO_4 + 5% H_2O_2 solution and contained 0.88 g U/L. The 2M NaCl scrub solution was contacted with the solvent for 5 minutes at an O/A ratio of 2.

Table 10

Screening of Uranium Stripping Agents

O/A	Stripping Agent	Separation Time	Comments
2/1	5% NaCl	64 sec	No stripping
2/1	13% $(NH_4)_2SO_4$	34 sec	Emulsion in aqueous phase
2/1	5% Na_2CO_3	3 min	Emulsion in aqueous phase
2/1	5% $NaHCO_3$	3 min	Precipitate in aqueous phase
2/1	13% NH_4Cl	66 sec	No stripping
2/1	5% Na_2SO_4	19 sec	Emulsion in aqueous phase
2/1	15% H_2SO_4	9 sec	No stripping
2/1	13% NH_4HCO_3	3 min	Emulsion in aqueous phase
3/1	0.5M $(NH_4)_2SO_4$	3 min	Emulsion in aqueous phase
3/1	0.5M $(NH_4)_2SO_4$ (pH 3 with H_2SO_4)	3 min	Emulsion in aqueous phase
3/1	0.5M $(NH_4)_2SO_4$ (pH 9 with NH_4OH)	3 min	Emulsion in aqueous phase
3/1	0.5M $(NH_4)_2SO_4$ (pH 9 with NH_4HCO_3)	3 min	Emulsion in aqueous phase
3/1	10% $MgSO_4$	96 sec	No stripping
3/1	1% K_2SO_4	3 min	No stripping
3/1	5% NH_4NO_2	1 min	No stripping
3/1	5% $(NH_4)_2C_2O_4.H_2O$	3 min	Emulsion in aqueous phase
3/1	5% K_2HPO_4	3 min	Emulsion in aqueous phase
3/1	10% $(NH_4)_2SO_4$ + 5% H_2O_2	69 sec	Emulsion in aqueous phase
3/1	5% H_2SO_4 + 5% H_2O_2	11 sec	Fair stripping, no emulsion

Organic: 2.5% TOPO in Shell 140

Table 11

Stripping with 5% H_2SO_4 + 5% H_2O_2

O/A Ratio	Stripped Solvent(g/L)			Strip Solution(g/L)			% Stripped		
	U	Th	Fe	U	Th	Fe	U	Th	Fe
7/1	3.61	0.04	0.024	8.29	0.002	0.001	24.7	95.2	49.0
5/1	3.42	0.019	0.017	7.49	0.002	0.009	31.1	63.3	88.6
3/1	2.76	0.011	0.017	6.42	0.002	0.007	44.5	61.1	81.0
1/1	1.35	0.004	0.007	3.34	0.002	0.008	69.4	66.7	100
1/3	0.60	0.002	0.006	1.60	0.001	0.002	99.8	100	100
1/5	0.28	0.001	0.005	0.82	0.001	0.002	85.2	83.3	100

Solvent feed to stripping: 4.8 g U/L

Table 12

Scrubbing for Sulphate Removal
(2.5% TOPO)

U (g/L)			SO_4 (g/L)	Fe (g/L)
Before Scrub	After Scrub	In Scrub Raff	In Scrub Raff	In Scrub Raff
0.88	0.88	0.0004	0.30	0.0006

2.0M NaCl, O/A 2/1

Continuous Testing

To test the process on a larger scale and in a continuous flow system, a Bell Engineering mixer-settler was used. The circuit that was tested is shown diagramatically in Fig. 6[6].

The circuit used 2.5% TOPO in Shell 140, and the leach feed analysed, in g/L: U = 0.70; Th = 0.25; Fe = 2.09 and Ra = 23,300 pCi/L. The extraction isotherm (Fig. 4) suggested that 5 stages would be needed to bring the raffinate to a U concentration of .005 g/L. The circuit, with 5 stages, was started at an A/O ratio of 3.2/1 (192 mL/60 mL per minute as flowrates).

The first 4-hour run showed higher than 99% extraction, with raffinate at less than 0.0002 g/L U. Extraction remained high throughout with the final raffinate showing no higher than 0.0002 g/L U as the phase ratio was increased to A/O 4.5/1 after 8 hours. The composite loaded organic contained 2.77 g/L U, 0.071 g/L Fe and .0006 g/L Th.

There were 2 stages of scrubbing with 15 g/L U at pH 0.5 at O/A = 2/1, followed by 7 stages of stripping with 5% H_2SO_4 + 5% H_2O_2. Stripping was at O/A = 3/1. To ensure sufficient sulphate scrubbing, 5 stages were used with 2 M HCl at O/A 3/1.

The strip raffinate sent for U precipitation contained 10.4 g/L U, .023 g/L Th, and .005 g/L Fe. After final scrubbing, the recycled organic contained 0.88 g/L U, $<$ .001 g/L Th and .0006 g/L Fe.

Discussion and Conclusion

The possibility of using chloride technology for the treatment of uranium-bearing ores and concentrates is enhanced with the ability to readily extract the uranium with TOPO. However the TOPO retains the uranium strongly and could only be stripped by using a mixture of H_2SO_4 + H_2O_2. The mechanism of the stripping is not well understood, but it is thought that the peroxide helps to solubilize the uranyl ion so that the H_2SO_4 can remove it from the organic phase. Obviously further chemical studies would be required to fully elucidate the stripping process chemistry. The strip solution can be easily precipitated to recover the uranium as uranyl peroxide. By raising the pH, the H_2O_2 is destroyed and

uranyl peroxide is formed. A 10% MgO slurry was added to the strip solution to raise the pH from 0.22 to pH 3.5 (optimum pH for precipitation lies in the range of 3.5 - 4.5[7,8]). After 4 hours precipitation time, 1 g/L of Percol 455 flocculant was added to settle the precipitate. Washing the precipitate with water was followed by drying.

The results have thus indicated a process to obtain high recovery of uranium from chloride leach liquors using TOPO. A unique stripping mixture enables good stripping effectiveness and the tests in the continuous circuit have indicated the technical feasibility of the process.

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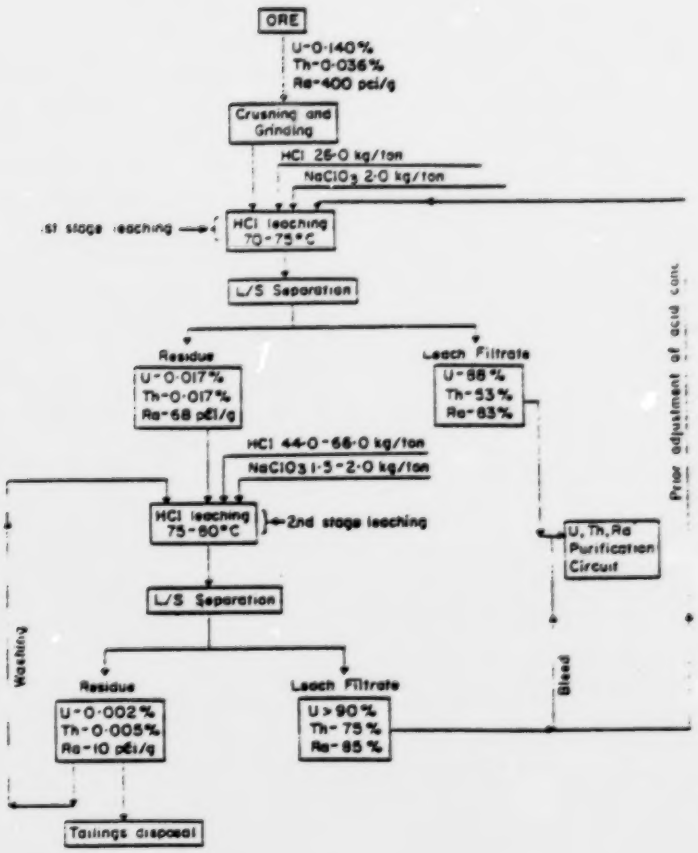

Figure 1. Hydrochloric Acid Leach Flowsheet.

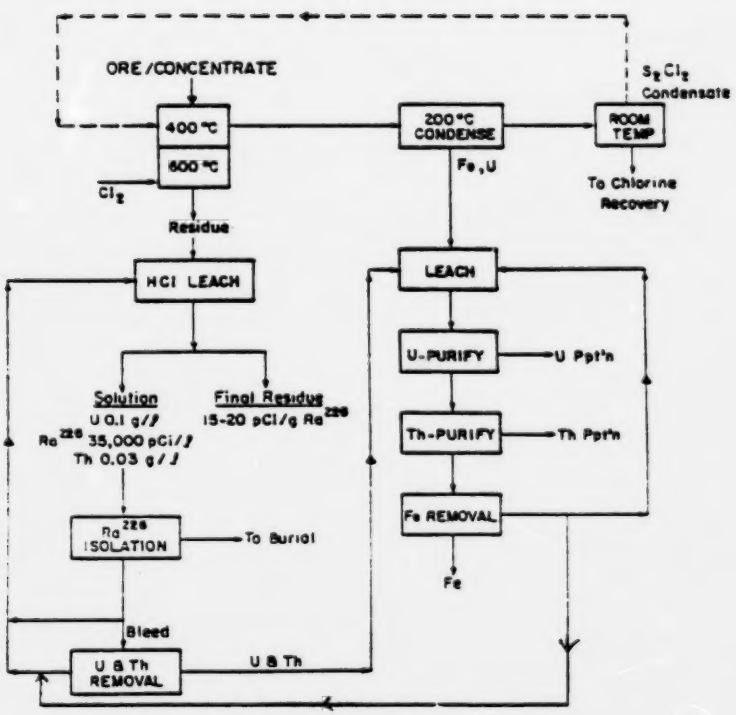

Figure 2. Chlorination Roast Flowsheet.

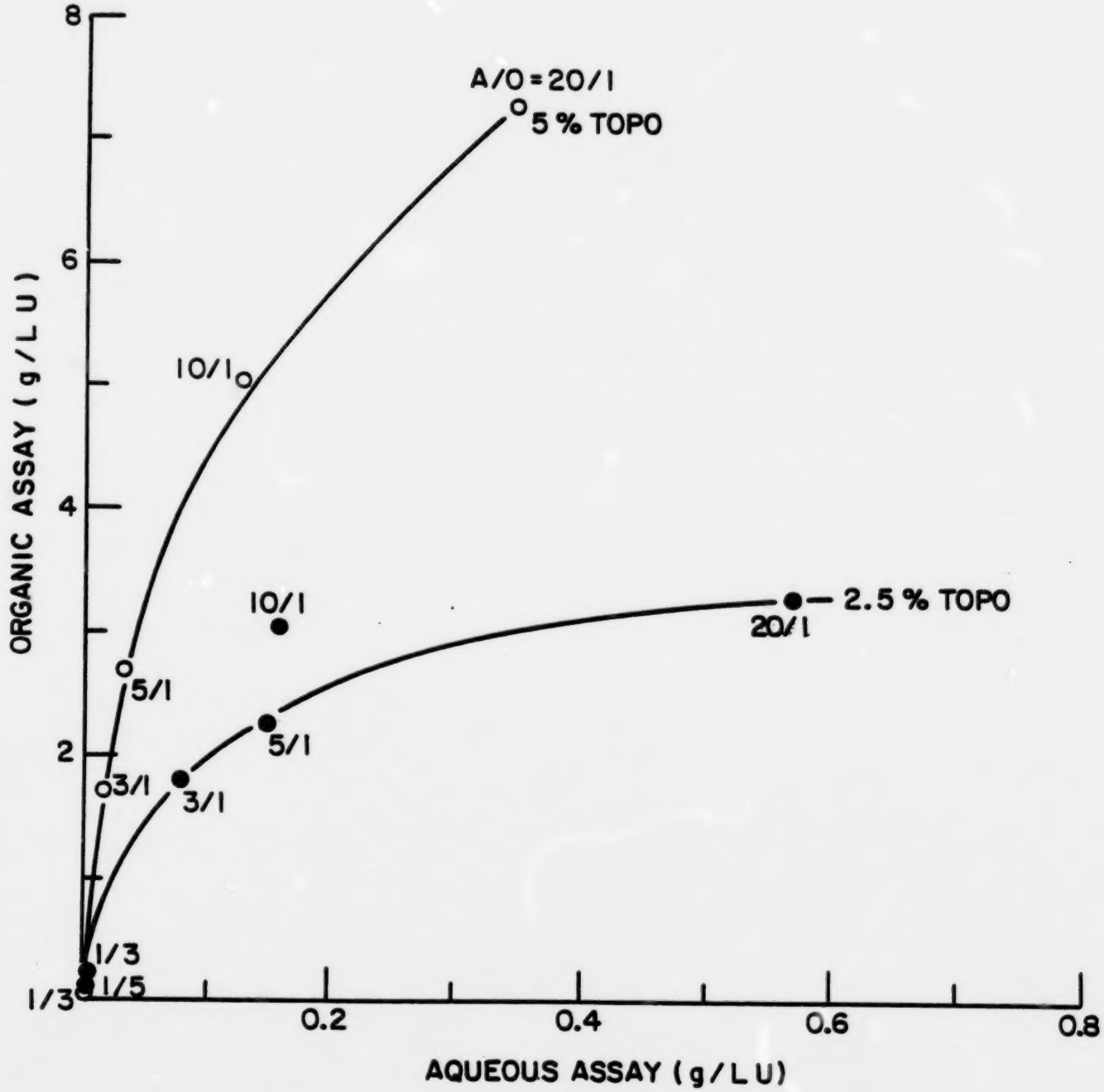

Figure 3. Extraction of Uranium With TOPO in Shell 140.

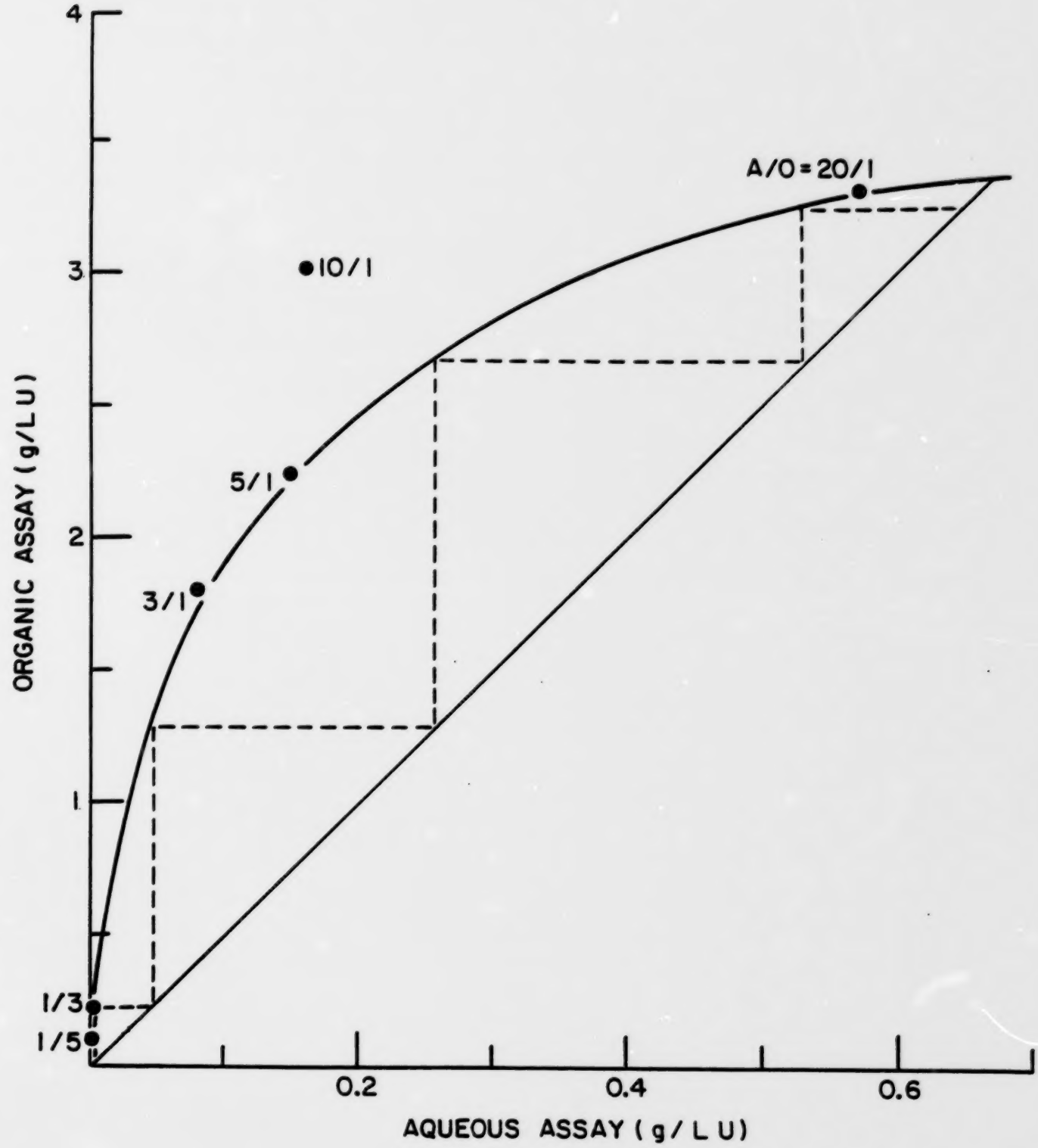

Figure 4. McCabe-Thiele Diagram for Uranium Loading 2.5% TOPO in Shell 140.

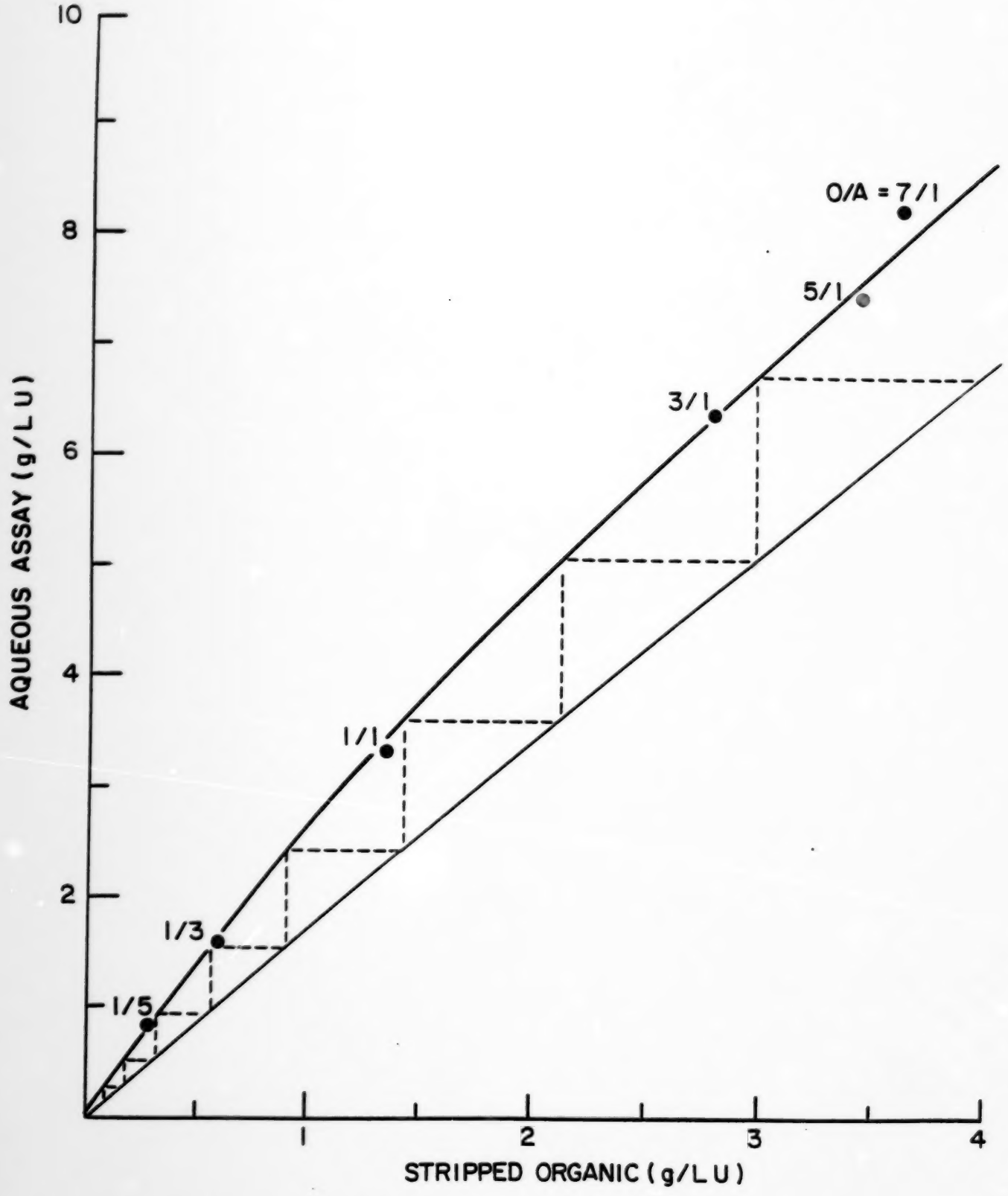

Figure 5. McCabe-Thiele Plot of Uranium Stripping
5% H_2SO_4 + 5% H_2O_2.

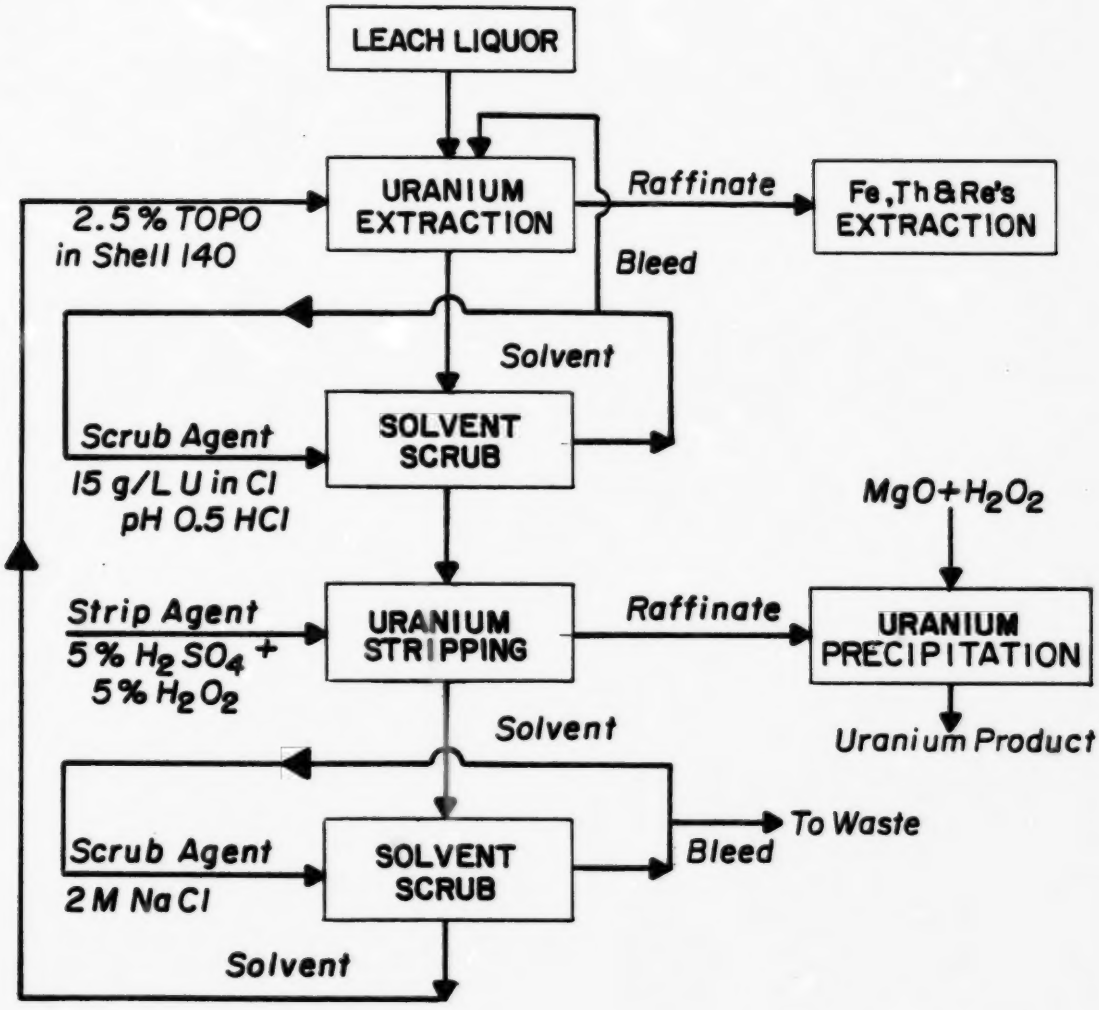

Figure 6. Schematic Flowsheet.